Table 20.3-A
Metals Analysis by ICP/MS: SW 846-6020A
Internal Standard Criteria for CCV, CCB and samples

Samples and QC samples	Isotope	Ref IS	Lower %	Upper %
Li	7	Sc	-	-
Be	9	Sc	-	-
B	11	Sc	-	-
Na	23	Y	-	-
Mg	24	Y	-	-
Al	27	Y	-	-
Si	28	Y	-	-
K	39	Y	-	-
Ca	44	Y	-	-
Sc (IS)	45	-	70	-
Ti	47	Y	-	-
V	51	Y	-	-
Cr	53	Y	-	-
Mn	55	Y	-	-
Fe	56	Y	-	-
Co	59	Y	-	-
Ni	60	Y	-	-
Cu	63	Y	-	-
Zn	66	Y	-	-
As	75	Y	-	-
Se	82	Y	-	-
Sr	87	Y	-	-
Y (IS)	89	-	70	-
Mo	98	Y	-	-
Ag	107	In (2), Y (3)	-	-
Cd	111	In	-	-
In (IS)	115	-	70	-
Sn	118	In	-	-
Sb	121	In (2), Y (3)	-	-
Ba	135	In	-	-
Tl	203	Bi	-	-
Pb	208	Bi	-	-
Bi (IS)	209	-	70	-
Th	232	Bi	-	-
U	238	Bi	-	-

Table 20.3-B Metals Analysis by ICP/MS: Method 200.8 Internal Standard Criteria for CCV, CCB and samples				
Samples and QC samples	Isotope	Ref IS	Lower %	Upper %
Li	7	Sc	-	-
Be	9	Sc	-	-
B	11	Sc	-	-
Na	23	Y	-	-
Mg	24	Y	-	-
Al	27	Y	-	-
Si	28	Y	-	-
K	39	Y	-	-
Ca	44	Y	-	-
Sc (IS)	45	-	60	125
Ti	47	Y	-	-
V	51	Y	-	-
Cr	53	Y	-	-
Mn	55	Y	-	-
Fe	56	Y	-	-
Co	59	Y	-	-
Ni	60	Y	-	-
Cu	63	Y	-	-
Zn	66	Y	-	-
As	75	Y	-	-
Se	82	Y	-	-
Sr	87	Y	-	-
Y (IS)	89	-	60	125
Mo	98	Y	-	-
Ag	107	In (2), Y (3)	-	-
Cd	111	In	-	-
In (IS)	115	-	60	125
Sn	118	In	-	-
Sb	121	In (2), Y (3)	-	-
Ba	135	In	-	-
Tl	203	Bi	-	-
Pb	208	Bi	-	-
Bi (IS)	209	-	60	125
Th	232	Bi	-	-
U	238	Bi	-	-

Table 20.4
Summary of Calibration and QC Procedures for Method 200.8 & 6020A

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]
ICPMS tuning sample.	Prior to initial calibration and calibration verification.	RSD < 5%. Amu +/- 0.1 true value.	Retune instrument then reanalyze tuning solution.
Initial calibration (minimum 4 standards and a blank).	Daily initial calibration prior to sample analysis.	r > 0.998.	N/A.
Initial Calibration verification (second source).	Daily after initial calibration,	All analytes within ±10% of expected value.	Correct problem and repeat initial calibration.
Calibration blank.	Before beginning a sample run, after every 10 samples and at end of the analysis sequence.	No analytes detected > 3 x IDL.	Correct problem then analyze calibration blank and previous 10 samples.
Calibration verification (Instrument Check Standard).	Before beginning a sample run, after every 10 samples and at the end of the analysis sequence.	All analyte(s) within ±10% of expected value.	Correct problem then repeat calibration and reanalyze all samples since last successful calibration.
Demonstrate ability to generate acceptable accuracy and precision using four replicate LCS analyses.	Once per analyst.	All analyte(s) within ± 20% of the expected value.	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria.
Method blank.	One per preparation batch.	NPW/SCM: No analytes detected > 3 x MDL. Drinking Water: No analytes detected > 2.2 x MDL.	Correct problem, re-digest and analyze method blank and all samples processed with the contaminated blank.

Table 20.4
Summary of Calibration and QC Procedures for Method 200.8 & 6020A

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]
Interference check solutions (ICS-A and ICS-AB).	At the beginning of an analytical run and every 8 hours.	ICS-A: All non-spiked analytes < ½ MQL; Spiked analytes within ±20% of true value. ICS-AB: Within ±20% of true value.	Terminate analysis; locate and correct problem; reanalyze ICS; reanalyze all affected samples.
LCS for the analyte.	One LCS per preparation batch.	All analytes within ± 15% of the expected value for 200.8 and +/- 20% for 6020A.	Correct problem, re-digest and reanalyze the LCS and all samples in the affected preparation batch.
Dilution test.	Each preparatory batch.	5X dilution must agree within ±10% of the original determination for analytes present at concentrations > 100x concentrations found in reagent blank.	Perform post digestion spike addition for failed analytes.
Post digestion spike addition.	When dilution test fails.	Recovery within 80%-120% of expected results.	Dilute the sample; reanalyze post digestion spike addition.
MS/MSD	5% frequency for 6020A, 10% frequency for 200.8.	QC advisory acceptance criteria, 70% - 130% for 200.8. 75% - 125% for 6020A.	Describe in Laboratory Review Checklist.
Internal Standards (ISs).	Every sample.	Sample IS intensity: *SW 846-6020a samples must meet >70% criteria. EPA 200.8 samples must meet 60-125% criteria.*	Perform corrective action and/or dilution and reprocess all effected samples.
MDL study.	Performed Annually	Detection limits established shall be < 1/3 the MQLs in Tables 21.1	None.

Table 20.4
Summary of Calibration and QC Procedures for Method 200.8 & 6020A

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]
IDL study.	Performed Quarterly	Average of standard deviation of reagent blank analyzed 7 times on at least 3 non-consecutive days.	None.
Low-level Initial Calibration Verification (LLICV)	Performed daily after Initial calibration	70%-130% of expected value spike at MQL.	Correct problem and repeat initial calibration.
Low-level Continuing Calibration Verification (LLCCV)	Performed before analysis of samples and after every 10 samples in the sequence.	70%-130% of expected value spike at MQL.	Correct problem then repeat calibration and reanalyze all samples of similar concentration since last successful calibration verification.
Low-level Quality Control Sample (LLQC)	One LLQC per quarter.	70%-130% of expected value spike at MQL. Carried through entire preparation process.	Correct problem, re-digest and reanalyze. If problem cannot be corrected, spike at a higher concentration and update PQLs accordingly.

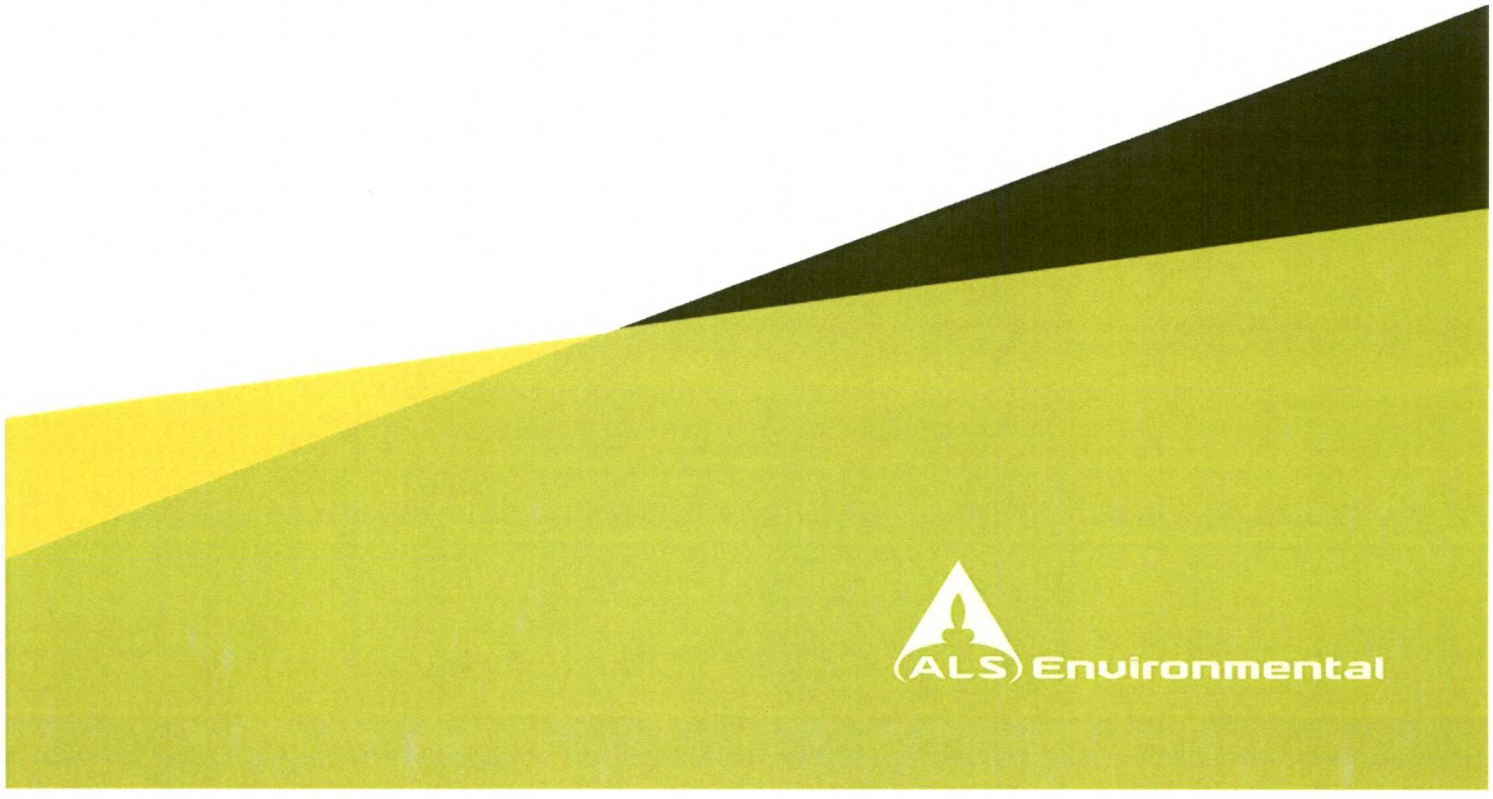
Attachment 21.10 - Interference Correction Equations

Interference Equation

Mass	Equation
51 :	(51)*1 - (53)*3.127 + (52)*0.353351
75 :	(75)*1 - (77)*3.127 + (82)*2.548505
82 :	(82)*1 - (83)*1.009
98 :	(98)*1 - (99)*0.146
111 :	(111)*1 - (108)*1.073
115 :	(115)*1 - (118)*0.016
208 :	(208)*1 + (206)*1 + (207)*1

ALS Standard Operating Procedure

DOCUMENT TITLE:	METALS SOLIDS DIGESTION
REFERENCED METHOD:	SW846 3050B
SOP ID:	HN-MET-009
REV. NUMBER:	R07
EFFECTIVE DATE:	09/15/2016



METALS SOLIDS DIGESTION

SW846 3050B

SOPID:	HN-MET-009	Rev. Number:	R07	Effective Date:	09/15/2016

Approved By: M. Lanning Date: 8/30/16
Department Supervisor

Approved By: Joseph Ryan Date: 8/29/16
Operations Manager

Approved By: Date: 8/29/16
QA Manager

Approved By: Date: 8/30/16
Laboratory Director

Archival Date: _____ Doc Control ID#: _____ Editor: _____

PROCEDURAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR 24 ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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METALS SOLIDS DIGESTION

1) Scope and Applicability

1.1 The objective of this method is to provide a general procedure for the preparation of soil or solid waste samples for determination of metals by ICP-MS or ICP-AES. The method is applicable to total leachable metals with the exception of mercury.

2) Summary of Procedure

2.1 A known amount of sample is digested with acid and peroxide by refluxing the sample in a hot block digester. The digestate is filtered (or allowed to settle), diluted to a specific volume, and analyzed by ICP-MS or ICP-AES. The procedure is based upon SW-846 Method 3050B.

3) Definitions

3.1 Preparation Batch: A grouping of 20 or less client samples processed under the same conditions, within an 8 hour working shift.

3.2 Laboratory Control Sample (LCS): An analyte-free matrix spiked with known concentrations of all target analytes. This is used to evaluate and document laboratory method performance.

3.3 Matrix: The component or substrate (e.g., surface water, groundwater, soil) which contains the analyte of interest.

3.4 Matrix Spike (MS): An aliquot of background sample spiked with a known concentrations of all target analytes. The spiking occurs prior to sample preparation and analysis. A matrix spike is used to assess the bias of a method in a given sample matrix.

3.5 Matrix Spike Duplicate (MSD): A duplicate aliquot of the background sample spiked with a known concentrations of all target analytes. Spiking occurs prior to sample preparation and analysis. The MS/MSD pair are used to assess precision and bias of a method in a given sample matrix.

3.6 Method Blank: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

3.7 HCl = Hydrochloric acid

3.8 H_2O_2 = Hydrogen peroxide

3.9 HNO_3 = Nitric acid

3.10 Limit of Quantitation (LOQ): The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ is also referred to as the method quantitation limit (MQL) or the reporting limit (RL).

3.11 Limit of Detection (LOD): an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory-dependent.

3.12 Method Detection Limit (MDL) study: the procedure, as described in 40CFR part 136, for determining the LOD based on statistical analysis of 7 low-level replicate spikes.

4) Health and Safety Warnings

4.1 Lab Safety: Due to various hazards in the laboratory, safety glasses and laboratory coats or aprons must be worn at all times while in the laboratory. In addition, gloves and a face shield should be worn when dealing with toxic, caustic, and/or flammable chemicals.

4.2 Chemical Hygiene: The toxicity or carcinogenicity of each reagent used has not been precisely defined; however, each chemical used should be treated as a potential health hazard. Exposure to laboratory reagents should be reduced to the lowest possible level. The laboratory maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets (MSDS) is available to all personnel involved in these analyses.

4.3 Waste Management: The principal wastes generated by this procedure are the method-required chemicals and standards. It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is required. Laboratory procedures in SOP HN-SAF-001, Waste Disposal Procedures, must be followed.

4.4 Pollution Prevention: The materials used in this method pose little threat to the environment when recycled and managed properly. The quantities of chemicals purchased should be based on the expected usage during its shelf life. Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards or reagents to be disposed.

5) Cautions

5.1 To prevent contamination of the analytical system, all supplies and materials coming in contact with the samples and instrument must be pre-cleaned in 1:4 HNO_3. This step may be omitted for purchased materials of known cleanliness.

5.2 Proper use and maintenance of pipettes is important to achieve good technique and obtain good LCS, MS, and MSD recoveries. Slow addition of H_2O_2 is critical.

5.3 Use all appropriate personal protective equipment when handling concentrated acids. This includes gloves, lab-coat, and a face shield at a minimum.

5.4 Samples will emit hazardous/noxious fumes upon addition of acid. Perform acid addition and sample digestion in a hood with adequate ventilation.

6) Interferences

6.1 Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material should be processed in accordance with the quality control requirements given in the Quality Control section to aid in determining whether this method is applicable to a certain waste.

7) Personnel Qualifications and Responsibilities

7.1 General Responsibilities - This method is restricted to use by or under the supervision of analysts experienced in the method.

7.2 Analyst - It is the responsibility of the analyst(s) to:

7.2.1 Each must read and understand this SOP and follow it as written. Any deviations or non-conformances must be documented and submitted to the QA Manager for approval.

7.2.2 Produce method compliant data that meets all quality requirements using this procedure and the Data Reduction, Review and Validation SOP (HN-QS-009).

7.2.3 Complete the required initial demonstration of proficiency before performing this procedure without supervision.

7.2.4 Complete an ongoing demonstration of proficiency annually when continuing to perform the procedure.

7.2.5 The analysts must submit data for peer or supervisor review.

7.3 Section Supervisor - It is the responsibility of the section supervisor to:

7.3.1 Ensure that all analysts have the technical ability and have received adequate training required to perform this procedure.

7.3.2 Ensure analysts have completed the required initial demonstration of proficiency before performing this procedure without supervision.

7.3.3 Ensure analysts complete an ongoing demonstration of proficiency annually when continuing to perform the procedure.

7.3.4 Ensure analysts produce method compliant data that meet all quality requirements using this procedure and the Data Reduction, Review and Validation SOP.

7.4 Project Manager - It is the responsibility of the Project Manager to ensure that all method requirements for a client requesting this procedure are understood by the laboratory prior to initiating this procedure for a given set of samples.

7.5 QA Manager: The QA Manager is responsible for

7.5.1 Approving deviations and non-conformances

7.5.2 Ensuring that this procedure is compliant with method and regulatory requirements,

7.5.3 Ensuring that the analytical method and SOP are followed as written through internal method and system audits.

8) Sample Collection, Handling, and Preservation

8.1 Soil samples are collected in 4 oz wide mouth glass containers. Refrigerated storage is not required. Should mercury analysis be required, soil samples must be refrigerated at 4°C prior to sample preparation.

8.2 Digestates do not require refrigeration.

8.3 The holding time is 180 days for soils.

9) Equipment and Supplies

9.1 Analytical Balance (capable of weighing to nearest 0.001 gram).

9.2 Hot Block digester capable of maintaining a temperature of 95°C.

9.3 50 mL digestion vessels, certified clean

9.4 Disposable watchglasses

9.5 Appropriately sized Class A volumetric flasks

9.6 Auto-pipettes, various delivery volumes

9.7 Filter mate®- 0.45µm push filter

9.8 Teflon® Chips, used as simulated solid matrix

10) Standards and Reagents

10.1 Acids used in the preparation of standards and for sample processing must be of high purity. Trace metal grade is recommended.

10.2 Concentrated HNO_3 (Trace metal grade)

10.3 Concentrated HCl (Trace metal grade)

10.4 H_2O_2 (30 %) - (Un-stabilized Trace metal grade, if available)

10.5 LCS and MS/MSD spiking solution (NIST traceable):

10.5.1 A 27 element standard is used each at 10 ppm, with the exception of the minerals (Fe, Ca, Mg, Na, and K at 1000 ppm) and Boron at 50 ppm. (Available from VHG, Custom Standard 901)

10.5.2 Ti Standard @ 10 ppm and Si Standard @ 50 ppm:

10.5.2.1 Prepare using NIST traceable 1000 ppm Ti and Si stock standards.
10.5.2.2 In a 500 ml volumetric flask, add 5 ml Ti stock standard, 25 ml Si stock standard, 10 ml HNO_3 and 5 ml HCl to 300 ml DI water.
10.5.2.3 Bring to final volume of 500 ml with DI.

10.5.3 LCS/MS/MSD Soil Spiking:

10.5.3.1 A 500 µl volume of each spike solution (10.5.1 and 10.5.2) is added to 1.00 gram of solid matrix that has been transferred to the digestion vessel.
10.5.3.2 Spiking of the sample shall be performed prior to the addition of any reagents.
10.5.3.3 Following digestion, the digestate is brought to a final volume of 50 ml and filtered or allowed to settle.

10.6 LLQC Spiking solution (NIST traceable):

10.6.1 Low-level Metals Mix Standard I w/ As, Ba, Cr, Co, Cu, Pb, Mn, Ni, Se, Ag, Sr, Tl, and V @ 0.5 mg/L **and** Be and Cd @ 0.2 mg/L **and** Al, Li, and Zn @ 1.0 mg/L **and** B @ 2.0 mg/L **and** Fe @ 8.0 mg/L **and** Mg, K, and Na @ 20 mg/L **and** Ca @ 50 mg/L. (available from VHG ZALSLAB1103-100 or equivalent)

10.6.2 Low-level Metals Mix Standard II w/ Sn @ 0.2 mg/L **and** Sb, Mo, and Ti @ 0.5 mg/L. (available from VHG ZALSLAB1104-100 or equivalent)

10.6.3 LLQC Soil Spike

10.6.3.1 Add 500 µl of each solution (10.6.1 and 10.6.2) to 1.00 gram of solid matrix that has been transferred to the digestion vessel.

10.6.3.2 Spiking of the sample shall be performed prior to the addition of any reagents.

10.6.3.3 Following digestion, the digestate is brought to a final volume of 50ml then filtered or allowed to settle.

11) Method Calibration

11.1 Perform support equipment (balances, etc.) calibration checks as required for daily use.

12) Sample Preparation/Analysis

12.1 Homogenize sample using formal subsampling techniques SOP (HN-QS-008), making sure to decant any standing water on top of the sample prior to processing.

12.2 Weigh (to the nearest 0.001 g) a 0.500g - 1.000g portion of sample into a labeled 50 ml digestion vessel.

12.2.1 For MBLK, LCS, and LLQC weigh out ~ 1.000g Teflon® chips.

12.2.2 For MS/MSD, parent samples, and duplicates, weigh out, as close as possible, equivalent masses in order to provide consistent calculations by LIMS.

12.3 Spike all QC samples prior to the addition of reagents.

12.4 Add 5 ml DI water, 5 ml concentrated HNO_3, and 1 ml concentrated HCl to the digestion vessel, mix the slurry, and cover with a ribbed watch glass. (Note: HCl is added prior to digestion to keep Ag in solution and preserve acceptable recovery.)

12.5 Reflux samples in the hot block at 90° – 95° C. Record digestion temperature in the associated logbook. Do not allow samples to boil.

12.5.1 Note: If brown fumes are observed during digestion, add an additional 5 ml of concentrated HNO_3 to the vessel and cover with a ribbed watch glass; heat until volume is reduced to approximately 5 ml.

12.6 Allow samples to digest for 45 minutes, then remove samples and cool. (Maintain a volume of at least 5 ml using DI).

12.7 Add 1.5 ml of 30% hydrogen peroxide (H_2O_2). Cover with the ribbed watch glass and return the vessels to the hot block for warming, to start the peroxide reaction. **Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence.**

12.8 Continue to add H_2O_2 in 1 ml aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. (NOTE: do not add more than a total of 5 ml of H_2O_2.)

12.9 Cover the digestion vessel and return to Hotblock under partial heat until volume is reduced to approximately 5 ml.

12.10 Remove samples from the block and cool.

12.11 Upon completion of the digestion, bring to a final volume of 50 ml using DI water and cap. Shake the capped digestates to homogenize acid and water layers.

12.12 Filter the digestate using a Filtermate® filter, or allow particulates to settle prior to analysis.

12.13 Appropriately label all storage containers. The label must include the sample ID and Prep Batch ID.

12.14 A sample batch is all samples of the same matrix, digested by the same method on a unit basis. A sample batch cannot exceed 20 samples. If more than 20 samples require analysis, the first 20 shall be the first batch, the next 20 shall be the second batch, and so forth.

13) Troubleshooting

13.1 Refer to determinative method for guidance.

14) Data Acquisition

14.1 Sample preparation data recorded in preparation logbooks is entered into LIMS for later use in analytical and QC calculations. LIMS assigns a prep batch number for the data entered. Record the LIMS prep batch number in the prep log.

15) Calculation, and Data Reduction Requirements

15.1 LIMS uses prep batch information including initial sample weight and final volume to perform calculations after analysis has been completed.

16) Quality Control, Acceptance Criteria and Corrective Action

16.1 Method Blank (MBLK): The method blank is a clean solid matrix (Teflon® chips) containing the same reagent percentages and processed as a sample. The method blank is used to verify the absence of bias in analytical results due to the laboratory reagents.

16.1.1 Frequency: One per batch (≤ 20 samples) of sample digestions.
16.1.2 Criteria: Refer to relevant section in the determinative method SOP (HN-MET-008 or HN-MET-015).

16.2 Laboratory Control Sample:

16.2.1 The LCS is prepared by adding 500 µL of the NIST traceable standard (section 10.5.3) to ~1.000g Teflon chips and processing as a sample.
16.2.2 Frequency: One LCS per batch of sample extractions. The results of the LCS are used for determining acceptability of results.
16.2.3 Criteria: Refer to the relevant section of the determinative method SOP (HN-MET-008 or HN-MET-015).

16.3 Matrix Spike/Matrix Spike Duplicate:

16.3.1 The matrix spike is prepared by adding 500 µL of NIST traceable standard (section 10.5.3) to the sample labeled as the matrix spike and matrix spike duplicate. The sample is then processed.
16.3.2 Frequency: Matrix spikes will be analyzed on a frequency of one spike set for each 20 samples analyzed. If fewer than 20 samples are in a batch, at least one spike set will be included.
16.3.3 Criteria: Refer to the relevant section of the determinative method SOP (HN-MET-008 or HN-MET-015).

16.4 Sample Duplicate

16.4.1 Frequency: Sample duplicates will be analyzed on a frequency of one for each 20 samples analyzed on a matrix specific basis. If fewer than 20 samples are in a batch, at least one duplicate will be included.
16.4.2 A matrix spike duplicate may be substituted for the duplicate analysis.
16.4.3 Criteria: Refer to the relevant section of the determinative method SOP (HN-MET-008 or HN-MET-015).

16.5 Low-Level Quality Control sample (LLQC):

16.5.1 The LLQC is prepared by adding 500 µL of NIST traceable standard (section 10.6.3) to ~1.000g Teflon chips and processing as a sample.
16.5.2 Frequency: One LLQC sample extracted per quarter. The results of the LLQC are used for determining ongoing performance of the method at low concentrations.
16.5.3 Criteria: Refer to the relevant section of the determinative method SOP (HN-MET-008 or HN-MET-015).

16.6 Deviations and non-conforming events must be documented using a Nonconformance Corrective Action Report (NCAR) or as an Exception Report item on the laboratory review checklist. For mandatory QC failures (e.g. LCS), the NCAR must be submitted to the QA Manager via the NCAR database.

17) Data Records Management

17.1 All data is stored both electronically and hard copy for 10 years.

17.2 All analytical sequence IDs and standard preparation information must be recorded in the Run logbook. Hardcopy computer printouts of analytical sequences and raw data must be retained and initialed by the analyst (electronic initials are acceptable). To simplify standard tracking, analyst must attempt to use one lot of reagents and standards with each batch.

17.3 Complete all pertinent sections in the respective logbooks. If not-applicable then line out the section. "Z" out or "X" out all large sections of the worksheet that are not used. Make all corrections with single line through, date and initial. Make NO obliterations when manually recording data.

17.4 Logbooks are controlled. Never remove a page from a logbook. Completed logbooks are returned to the QA department when filled and no longer needed in the work area.

17.5 The effective date of this SOP is the date in the header or last signature date, whichever is most recent.

17.6 Logbooks must be reviewed monthly by the department supervisor.

17.7 Logbooks must be reviewed quarterly by the QA Staff.

18) Contingencies for Handling Out of Control Data

18.1 When method required QC exceedances occur, in every case where sample data quality are affected, the source of the QC exceedance must be determined, corrected and sample reanalysis carried out when possible.

18.2 When affected sample analysis can not be repeated due to limitations (i.e. sample availability, or if reanalysis can only be performed after expiration of a sample hold

time), the reporting of data associated with exceeded QC data must be appropriately flagged and narrated. This documentation is necessary to define for the data user the effect of the error has upon the data quality of the results reported (e.g. E flag data indicate the result to be only an estimate).

18.3 All analysts must report sufficient comments in laboratory data review checklist for exceeded QC associated with sample results so that project management can further narrate and ensure data qualifiers (flags) are properly assigned to the reported data.

18.4 NCARs must be issued for QC system exceedances. Matrix interferences are reported using the analyte reporting comment section in LIMS or using the Laboratory Data review checklist.

19) Method Performance

19.1 Initial Demonstration of Proficiency- Each analyst must perform an initial demonstration of proficiency on a method and matrix basis with a successful analysis of four LCS where acceptable precision and accuracy are generated. The accuracy component must fall within LCS criteria. The precision component must be less than 20% for duplicate RPD data.

19.2 Method Detection Limits (MDLs) must be determined on an annual basis (at minimum) or whenever major modifications are performed.

20) Summary of Changes

Table 20.1 Summary of Changes

Revision Number	Effective Date	Document Editor	Description of Changes
R04	7/1/12	CES	Formatting/Compliance
R05	10/1/2013	CES	Formatting; Addition of dry-spiking requirement
R06	1/31/16	CES	Included ICP-AES analysis. Updated document revision and data retention requirements.
R07	9/15/16	CES	Section 12.1 added.
R07	9/15/16	CES	Section 12.5.1 revised to approx. 5mL final volume.
R07	9/15/16	CES	Section 12.9 revised to approx. 5mL final volume.

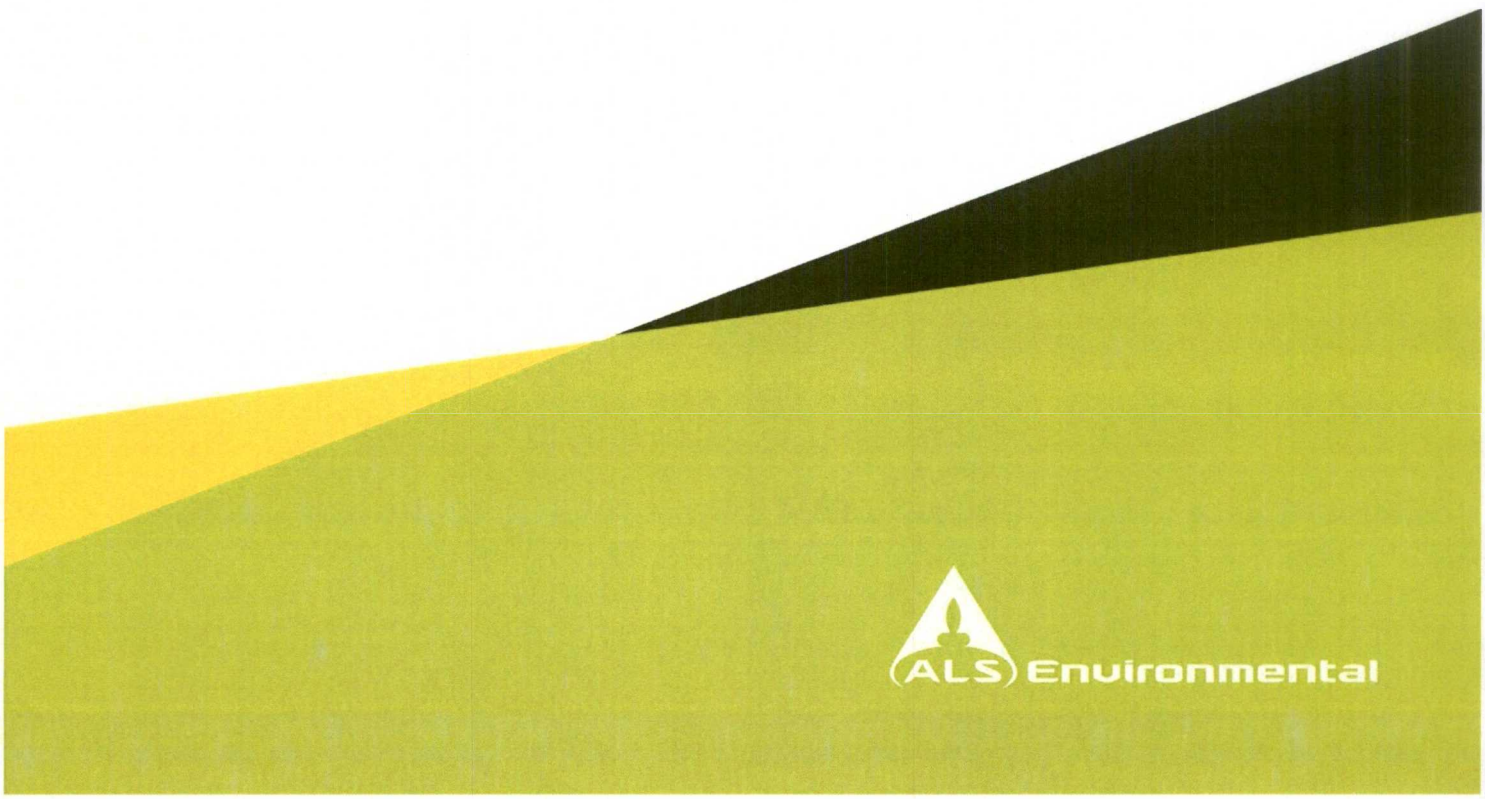
21) References and Related Documents

21.1 U.S. Environmental Protection Agency, "Method 3050B Acid Digestion of Sediments, Sludges and Soils", Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update III, June 13, 1997.

21.2 ALS Environmental Quality Assurance Manual, Revision (most current)

ALS Standard Operating Procedure

DOCUMENT TITLE:	METALS AQUEOUS DIGESTION
REFERENCED METHOD:	SW846 3005A / EPA 200.8
SOP ID:	HN-MET-010
REV. NUMBER:	R09
EFFECTIVE DATE:	12/31/2016



METALS AQUEOUS DIGESTION

SW846 3005A / EPA 200.8

SOPID:	HN-MET-010	Rev. Number:	R09	Effective Date:	12/31/2016

Approved By: M. Tanner Date: 12/21/16
Department Supervisor

Approved By: Joseph Rich Date: 12/21/16
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Approved By: Date: 12/21/16
QA Manager

Approved By: Date: 12/21/16
Laboratory Director

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Signature	Title	Date
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METALS AQUEOUS DIGESTION

1) Scope and Applicability

1.1 This method is applicable to the digestion of aqueous samples for determination of metals by ICP-MS or ICP-AES.

1.2 This method is applicable to a variety of aqueous matrices including drinking water, non-potable water, and TCLP/SPLP extracts.

2) Summary of Procedure

2.1 A mixture of nitric acid, hydrochloric acid, and the aqueous material to be analyzed is refluxed in a covered digestion tube in a hot block digester. This procedure is based upon SW-846 Method 3005A and EPA 200.8.

3) Definitions

3.1 Preparation Batch: Twenty or less client samples (excluding QC samples) processed under the same conditions, within an 8 hour working shift.

3.2 Laboratory Control Sample (LCS): An analyte-free matrix spiked with known concentrations of all target analytes. This is used to evaluate and document laboratory method performance.

3.3 Matrix: The component or substrate (e.g., surface water, groundwater, soil) which contains the analyte of interest.

3.4 Matrix Spike (MS): An aliquot of background sample spiked with a known concentrations of all target analytes. The spiking occurs prior to sample preparation and analysis. A matrix spike is used to assess the bias of a method in a given sample matrix.

3.5 Matrix Spike Duplicate (MSD): A duplicate aliquot of the background sample spiked with a known concentrations of all target analytes. Spiking occurs prior to sample preparation and analysis. The MS/MSD pair are used to assess precision and bias of a method in a given sample matrix.

3.6 Method Blank: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

3.7 HCl: Hydrochloric acid

3.8 HNO_3: Nitric acid

3.9 Limit of Quantitation (LOQ): The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ is also referred to as the method quantitation limit (MQL) or the reporting limit (RL).

3.10 Limit of Detection (LOD): an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory-dependent.

3.11 Method Detection Limit (MDL) study: the procedure, as described in 40CFR part 136, for determining the LOD based on statistical analysis of 7 low-level replicate spikes.

4) Health and Safety Warnings

4.1 Lab Safety: Due to various hazards in the laboratory, safety glasses and laboratory coats or aprons must be worn at all times while in the laboratory. In addition, gloves and a face shield should be worn when dealing with toxic, caustic, and/or flammable chemicals.

4.2 Chemical Hygiene: The toxicity or carcinogenicity of each reagent used has not been precisely defined; however, each chemical used should be treated as a potential health hazard. Exposure to laboratory reagents should be reduced to the lowest possible level. The laboratory maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets (MSDS) is available to all personnel involved in these analyses.

4.3 Waste Management: The principal wastes generated by this procedure are the method-required chemicals and standards. It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is required. Laboratory procedures in SOP HN-SAF-001, Waste Disposal Procedures, must be followed.

4.4 Pollution Prevention: The materials used in this method pose little threat to the environment when recycled and managed properly. The quantities of chemicals purchased should be based on the expected usage during its shelf life. Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards or reagents to be disposed.

5) Cautions

5.1 To prevent contamination of the samples, all supplies and materials coming in contact with the samples and instrument must be pre-cleaned in 1:4 HNO_3 or purchased and demonstrated as having a known level of cleanliness.

5.2 Do not clean applicable glassware with chromic acid reagents. Chromium is often an analyte of interest.

6) Interferences

6.1 Interferences are discussed in the applicable analytical SOP (HN-MET-008 or HN-MET-015).

6.2 Digestates containing suspended matter may require filtration upon completion of digestion in order to prevent clogging of the analytical instrumentation.

6.3 Samples high in solids or basic in nature may need dilution so not to impair the acidification process.

7) Personnel Qualifications and Responsibilities

7.1 General Responsibilities - This method is restricted to use by or under the supervision of analysts experienced in the method.

7.2 Analyst - It is the responsibility of the analyst(s) to:

7.2.1 Each must read and understand this SOP and follow it as written. Any deviations or non-conformances must be documented and submitted to the QA Manager for approval.

7.2.2 Produce method compliant data that meets all quality requirements using this procedure and the Data Reduction, Review and Validation SOP (HN-QS-009).

7.2.3 Complete the required initial demonstration of proficiency before performing this procedure without supervision.

7.2.4 Complete an ongoing demonstration of proficiency annually when continuing to perform the procedure.

7.2.5 The analysts must submit data for peer or supervisor review.

7.3 Section Supervisor - It is the responsibility of the section supervisor to:

7.3.1 Ensure that all analysts have the technical ability and have received adequate training required to perform this procedure.

7.3.2 Ensure analysts have completed the required initial demonstration of proficiency before performing this procedure without supervision.

7.3.3 Ensure analysts complete an ongoing demonstration of proficiency annually when continuing to perform the procedure.

7.3.4 Ensure analysts produce method compliant data that meet all quality requirements using this procedure and the Data Reduction, Review and Validation SOP.

7.4 Project Manager - It is the responsibility of the Project Manager to ensure that all method requirements for a client requesting this procedure are understood by the laboratory prior to initiating this procedure for a given set of samples.

7.5 QA Manager: The QA Manager is responsible for

7.5.1 Approving deviations and non-conformances

7.5.2 Ensuring that this procedure is compliant with method and regulatory requirements,

7.5.3 Ensuring that the analytical method and SOP are followed as written through internal method and system audits.

8) Sample Collection, Handling, and Preservation

8.1 Aqueous samples must be acidified to pH <2 with HNO_3.

8.1.1 Preservation may be completed in the field. However, to avoid hazards associated w/strong acids in the field; samples may be returned to the laboratory within two (2) weeks of collection and preserved upon receipt.

8.1.2 Samples preserved upon receipt must be placed on "hold" for a minimum of 24 hours and be rechecked for appropriate pH prior to proceeding with analysis.

8.1.3 Samples and digestates for metals analysis (except mercury) do not require refrigeration.

8.2 The holding time is 180 days.

9) Equipment and Supplies

9.1 Hot Block digester capable of maintaining a temperature of 90-95°C.

9.2 50 ml digestion tubes with watch glass and reflux cap (Env Express SC475 or equivalent)

9.3 Auto-pipettes (Eppendorf, Oxford, or equivalent) - various delivery volumes.

9.4 Filtering apparatus (as required)

10) Standards and Reagents

10.1 Acids used in the preparation of standards and for sample processing must be of high purity. Distilled acids are recommended.

10.2 Concentrated HNO_3 (Trace metal grade)

10.3 Concentrated HCl (Trace metal grade)

10.4 LCS and MS/MSD spiking solution (NIST traceable):

10.4.1 A 27 element standard is used each at 10 ppm, with the exception of the minerals (Fe, Ca, Mg, Na, and K at 1000 ppm) and Boron at 50 ppm. (Available from VHG, Custom Standard 901)

10.4.2 Ti Standard @ 10 ppm and Si Standard @ 50 ppm:

10.4.2.1 Prepare using NIST traceable 1000 ppm Ti and Si stock standards.

10.4.2.2 In a 500 ml volumetric flask, add 5 ml Ti stock standard, 25 ml Si stock standard, 10 ml HNO_3 and 5 ml HCl to 300 ml DI water.

10.4.2.3 Bring to final volume of 500 ml with DI.

10.4.3 LCS/MS/MSD Aqueous Spiking:

10.4.3.1 A 500 µl volume of each spike solution (10.4.1 and 10.4.2) is added to 50ml reagent water (LCS) or sample matrix (MS/MSD) that has been transferred to the digestion vessel.

10.4.3.2 Spiking must occur prior to the addition of any reagents.

10.4.3.3 Following digestion, the digestate is brought to a final volume of 50 ml and filtered or allowed to settle if particulate material is noted.

10.5 LLQC Spiking solution (NIST traceable):

10.5.1 Low-level Metals Mix Standard I w/ As, Ba, Cr, Co, Cu, Pb, Mn, Ni, Se, Ag, Sr, Tl, and V @ 0.5 mg/L **and** Be and Cd @ 0.2 mg/L **and** Al, Li, and Zn @ 1.0 mg/L **and** B @ 2.0 mg/L **and** Fe @ 8.0 mg/L **and** Mg, K, and Na @ 20 mg/L **and** Ca @ 50 mg/L. (available from VHG ZALSLAB1103-100 or equivalent)

10.5.2 Low-level Metals Mix Standard II w/ Sn @ 0.2 mg/L **and** Sb, Mo, and Ti @ 0.5 mg/L. (available from VHG ZALSLAB1104-100 or equivalent)

10.5.3 LLQC Aqueous Spike

10.5.3.1 Add 500 µl of each solution (10.5.1 and 10.5.2) to 50ml reagent water that has been transferred to the digestion vessel.

10.5.3.2 Spiking must occur prior to the addition of any reagents.

10.5.3.3 Following digestion, the digestate is brought to a final volume of 50ml.

10.5.3.4 Record LLQC preparation in the LIMS preparation sheet.

11) Method Calibration

11.1 Perform support equipment (balances, etc.) calibration checks as required for daily use.

12) Sample Preparation/Analysis

12.1 Samples for dissolved metals analysis may be digested or analyzed directly depending on client requirements. Regardless of whether the samples are digested, a filtered MBLK and LCS must accompany all samples within a filter batch. This QC is to be processed in the same manner as the samples. It is recommended that samples requiring digestion be filtered in a separate batch from those being analyzed directly.

12.2 Transfer a 50 ml representative aliquot of the well-mixed sample to a 50 ml digestion tube. Carefully add 2.0 ml of concentrated HNO_3 and 0.5 ml of concentrated HCl. Cover the tube with the open reflux cap and place the rack of tubes into the Hotblock.

12.3 Set the controller temperature to 115 - 120° C. (This setting will maintain a digestate temperature of ~ 95° C.) Record the actual sample digestion temperature in the digestion logbook.

12.4 Monitor the procedure to ensure that the digesting samples do not boil.

12.5 Concentrate to a volume of ~10 ml.

12.6 Cover the sample with a watchglass and allow to reflux for 30 minutes.

12.7 Do not allow digestates to reach dryness.

12.8 Remove and cool.

12.9 Rinse tube walls and reflux cap with ~ 20 ml of DI water.

12.10 Bring to a final volume to 50 ml with DI water. (The final acid concentration should be 4.0% HNO_3 and 1.0% HCl). If necessary, filter the sample to remove particulates with a 45 um filter. If filtration is used, the MBLK shall also be filtered to monitor for potential bias.

12.11 Prepare a method blank, LCS at a 5% frequency, and MS/MSD pair at a 5% frequency for Method 6020A/6010C and 10% frequency for EPA 200.8/200.7. A batch consists of 20 or less samples. (See Sections 10.4, 10.5, 16.3, 16.4, and 16.6)

13) Troubleshooting

13.1 Refer to determinative method for guidance.

14) Data Acquisition

14.1 Sample preparation data recorded in preparation logbooks must be entered into the LIMS for later use in analytical and QC calculations. LIMS assigns a prep batch number for the data entered. Record the LIMS prep batch number in the prep log.

15) Calculation, and Data Reduction Requirements

15.1 LIMS utilizes instrument measurements in conjunction with the digestion data to perform calculations and reporting after analysis has been completed.

16) Quality Control, Acceptance Criteria and Corrective Action

16.1 During the digestion procedure, samples must not be allowed to boil or go to dryness. If so, the digestion must be repeated.

16.2 Method Blank (MBLK):

16.2.1 Preparation:

16.2.1.1 The method blank is a digestion tube containing 50 ml of DI water, digested with the same reagents as an actual sample.

16.2.2 Frequency:

16.2.2.1 One per batch of sample digestions or every 20 samples (whichever is less).

16.2.3 Acceptance Criteria:

16.2.3.1 All analytes of interest must be less than the MDL.

16.2.4 Corrective Action:

16.2.4.1 If the method blank results do not meet the acceptance criteria above, the laboratory must take corrective action to locate and reduce the source of the contamination and re-digest and reanalyze all samples associated with the failed method blank.

16.2.4.2 If samples cannot be re-run because of insufficient sample, a non-conformance/corrective action report must be initiated and issued to project management and to the QA Manager. The NCR/CAR must provide sufficient detail for project narration and meet the requirements documented in HN-QS-003.

16.2.4.3 Data reported with an associated contaminated method blank must be flagged with a "B".

16.3 Laboratory Control Sample (LCS):

16.3.1 Preparation

16.3.1.1 Add 500 ul of the spiking solution (Section 10.4) to 50 ml of DI water and process as a sample.

16.3.2 Frequency:

16.3.2.1 One set per batch of sample digestions or every 20 samples (whichever is less).

16.3.3 Acceptance Criteria and Corrective Actions:

16.3.3.1 Refer to the relevant section in the determinative method.

16.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD):

16.4.1 Preparation

16.4.1.1 Add 500 ul of spiking solution (Section 10.4) to 50 ml of client sample and process as a sample.

16.4.2 Frequency:

16.4.2.1 Matrix spikes shall be prepared with each batch of client samples or every 10 samples (whichever is less) for EPA 200.8/200.7 and every 20 samples for Method 6020A/6010C.

16.4.3 Acceptance Criteria and Corrective Action:

16.4.3.1 Refer to the relevant section in the determinative method.

16.5 Sample Duplicate

16.5.1 Frequency:

16.5.1.1 A sample duplicate should be processed with each digestion batch.
16.5.1.2 A matrix spike duplicate may be substituted for the duplicate analysis unless required otherwise by project specifications.

16.5.2 Criteria and Corrective Actions:

16.5.2.1 Refer to the relevant section in the determinative method.

16.6 Low-Level Quality Control Sample (LLQC):

16.6.1 Preparation:

16.6.1.1 The LLQC is prepared by adding 500 µL of NIST traceable standard (section 10.5) to 50ml DI and processing as a sample.

16.6.2 Frequency:

16.6.2.1 One LLQC per quarter. The results of the LLQC are used for determining the ongoing acceptability of results at similar concentration.

16.6.3 Criteria and Corrective Actions:

16.6.3.1 Refer to the relevant section of the determinative method.

16.7 Deviations and non-conforming events must be documented using a Nonconformance Corrective Action Report (NCAR) or as an Exception Report item on the laboratory review checklist. For mandatory QC failures (e.g. LCS), the NCAR must be submitted to the QA Manager via the NCAR database.

17) Data Records Management

17.1 All data is stored both electronically and hard copy for 10 years.

17.2 All analytical sequence IDs and standard preparation information must be recorded in the Run logbook. Hardcopy computer printouts of analytical sequences and raw data must be retained and initialed by the analyst (electronic initials are acceptable). To simplify standard tracking, analyst must attempt to use one lot of reagents and standards with each batch.

17.3 Complete all pertinent sections in the respective logbooks. If not-applicable then line out the section. "Z" out or "X" out all large sections of the worksheet that are not used. Make all corrections with single line through, date and initial. Make NO obliterations when manually recording data.

17.4 Logbooks are controlled. Never remove a page from a logbook. Completed logbooks are returned to the QA department when filled and no longer needed in the work area.

17.5 The effective date of this SOP is the date in the header or last signature date, whichever is most recent.

17.6 Logbooks must be reviewed monthly by the department supervisor.

17.7 Logbooks must be reviewed quarterly by the QA Staff.

18) Contingencies for Handling Out of Control Data

18.1 When method required QC exceedances occur, in every case where sample data quality are affected, the source of the QC exceedance must be determined, corrected and sample reanalysis carried out when possible.

18.2 When affected sample analysis cannot be repeated due to limitations (i.e. sample availability, or if reanalysis can only be performed after expiration of a sample hold time), the reporting of data associated with exceeded QC data must be appropriately flagged and narrated. This documentation is necessary to define for the data user the effect of the error has upon the data quality of the results reported (e.g. E flag data indicate the result to be only an estimate).

18.3 All analysts must report sufficient comments in laboratory data review checklist for exceeded QC associated with sample results so that project management can further narrate and ensure data qualifiers (flags) are properly assigned to the reported data.

18.4 NCARs must be issued for QC system exceedances. Matrix interferences are reported using the analyte reporting comment section in LIMS or using the Laboratory Data review checklist.

19) Method Performance

19.1 Initial Demonstration of Proficiency- Each analyst must perform an initial demonstration of proficiency on a method and matrix basis with a successful analysis of four LCS where acceptable precision and accuracy are generated. The accuracy component must fall within LCS criteria. The precision component must be less than 20% for duplicate RPD data.

19.2 Method Detection Limits (MDLs) must be determined on an annual basis (at minimum) or whenever major modifications are performed.

19.3 A LLQC sample is processed quarterly to demonstrate acceptable recovery at the reporting limit.

20) Summary of Changes

Table 20.1 Summary of Changes

Revision Number	Effective Date	Document Editor	Description of Changes
R04	7/1/12	CES	Formatting/Compliance
R05	9/1/12	CES	Sec. 12.8 - removed LLQC; Sec. 16.6.2 - frequency to every quarter; add sec. 19.3.
R06	10/1/13	CES	Formatting; update spiking requirements
R07	10/1/14	CES	Addition of section 12.1.

R08	12/15/16	CES	Added section 1.3
R08	12/15/16	CES	Updated Section 14.1 for direct LIMS entry.
R08	12/15/16	CES	Updated document review and record retention criteria.
R09	12/31/16	CES	Section 12.5 updated to ~10 mL
R09	12/31/16	CES	Section 12.6 added for 30 min. reflux w/ watchglass

21) References and Related Documents

21.1 U.S. Environmental Protection Agency, "Method 3005A Acid Digestion of Waters for Total Recoverable and Dissolved Metals for Analysis by FLAA or ICP Spectroscopy", Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update III, June 13, 1997.

21.2 U.S. Environmental Protection Agency, "Method 200.8, Inductively Coupled Plasma – Mass Spectrometry," Methods for Chemical Analysis of Water and Wastes, Revision 5.4, 1994.

21.3 ALS Environmental Quality Assurance Manual, Revision (most current)

ALS Standard Operating Procedure

DOCUMENT TITLE:	METALS BY ICP-AES
REFERENCED METHOD:	EPA 200.7 / SW846 6010C
SOP ID:	HN-MET-015
REV. NUMBER:	R02
EFFECTIVE DATE:	08/31/2016

ALS Environmental

METALS BY ICP-AES

EPA 200.7 / SW846 6010C

SOPID:	HN-MET-015	Rev. Number:	R02	Effective Date:	08/31/16

Approved By: M. Hannix Date: 8/1/16
Department Supervisor

Approved By: Joseph Riley Date: 8/4/16
Operations Manager

Approved By: [signature] Date: 8/1/16
QA Manager

Approved By: [signature] Date: 8/1/16
Laboratory Director

Archival Date:	Doc Control ID#:	Editor:

PROCEDURAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR 24 ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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METALS BY ICP-AES

1) Scope and Applicability

1.1 Inductively coupled plasma-atomic emission spectroscopy (ICP-AES) is applicable to the determination of multiple elements in various matrices including drinking water, non-potable water, solid/chemical waste, biological tissues, and air/emissions.

1.2 When aqueous dissolved constituents are required, samples must be filtered and acid-preserved prior to analysis. No digestion is required prior to analysis. Acid digestion is required for groundwater, aqueous samples, industrial wastes, soils, sludges, sediments, and other solid wastes for which total (acid-leachable) elements are required.

1.3 SW-846 Method 6010C or US EPA 200.7 is used to determine the analytes listed in Attachment A. Additional elements may be added based upon an acceptable Demonstration of Capability (DOC) as well as completion of required method detection limit studies for aqueous and solid matrices.

1.4 Method Detection Limits (MDLs), Method Quantitation Limits (MQLs) and linear ranges will vary dependent upon the analyte, matrices, instrumentation, and operating conditions.

1.5 Use of this method is restricted to analysts who are knowledgeable in the recognition and in the correction of spectral, chemical, and physical interferences in ICP-OES.

2) Summary of Procedure

2.1 Prior to analysis, samples requiring total ("acid-leachable") values must be digested using appropriate sample preparation methods.

2.2 Method 6010C and 200.7 describe the multi-elemental determination of analytes by ICP-OES. The method measures characteristic emission spectra by optical spectrometry. Samples are nebulized and transported to the plasma torch. Element specific emission spectra are produced by interaction of the aerosol with the radio frequency inductively coupled plasma. Spectra are dispersed via a grating and intensities monitored by a photometric device.

2.3 Typical Method Quantitation Limits (MQL/MRL) for this method in the determination of individual elements range from 0.5 - 2.0 mg/kg for solid matrices and 5.0 - 20 ug/L for aqueous matrices. MQL/MRLs will be proportionately higher for sample extracts that require dilution. MQL/MRLs, and all other calculated concentrations, shall be based upon values obtained from sample extracts processed according to this SOP and the applicable extraction SOP.

3) Definitions

3.1 Laboratory Control Sample (LCS): An analyte-free matrix spiked with known concentrations of all target analytes. This is used to evaluate and document laboratory method performance.

3.2 Matrix: The component or substrate (e.g., surface water, groundwater, soil) which contains the analyte of interest.

3.3 Matrix Spike (MS): An aliquot of background sample spiked with a known concentrations of all target analytes. The spiking occurs prior to sample preparation and analysis. A matrix spike is used to assess the bias of a method in a given sample matrix.

3.4 Matrix Spike Duplicate (MSD): A duplicate aliquot of the background sample spiked with a known concentrations of all target analytes. Spiking occurs prior to sample preparation and analysis. The MS/MSD pair are used to assess precision and bias of a method in a given sample matrix.

3.5 Post Digestion Spike (PDS): A known amount of target analyte added to a sample extract following sample digestion and concentration.

3.6 Initial Calibration Verification (ICV): A second source standard utilized to verify the accuracy of the established initial calibration.

3.7 Method Blank: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

3.8 Limit of Quantitation (LOQ): The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ is also referred to as the method quantitation limit (MQL) or the reporting limit (RL).

3.9 Limit of Detection (LOD): an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory-dependent.

3.10 Method Detection Limit (MDL) study: the procedure, as described in 40CFR part 136, for determining the LOD based on statistical analysis of 7 low-level replicate spikes. The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

3.11 Standard Curve: A plot of concentrations of known analyte standards versus the instrument response to the analyte.

3.12 Internal Standard: A known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the analytical test method.

3.13 Interference Check Solution (ICS): A solution containing known concentrations of interfering elements.

3.14 Linear Dynamic Range (LDR): The concentration range through which the instrument response is linear.

3.15 Low-Level Quality Control sample (LLQC): A clean matrix sample spiked at the MQL and carried through the entire preparation and analysis process.

3.16 Continuing Calibration Blank (CCB): Acidified de-ionized water containing the same concentration of acids utilized in the standards and samples.

3.17 Continuing Calibration Verification (CCV): An acidified solution containing elements at or near the mid-point of the calibration curve.

3.18 Low-Level Initial Calibration Verification (LLICV): A sample spiked at the MQL, used to validate the lower end of the initial calibration.

3.19 Low-Level Continuing Calibration Verification (LLCCV): A sample spiked at the MQL and analyzed periodically throughout an analytical sequence, monitoring continued performance of the lower end of a calibration.